Istituto Nazionale di Fisica Nucleare

Laboratori Nazionali del Gran Sasso

DarkSide Project Process Procedure

UAr Implementation

Process Procedure Number: DS-Oper 11 - Rev. 3

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Procedure validity: from Revision Date
to End of Project

1. Table of Contents

2. Revision History

Revision #	Date	Author(s)	Rationale	Sections Updated
1	Mar, 6 2015	CK, AG, FG	First emission	All
2	Mar, 9 2015	FG	Second emission	§8 Procedure
3	Mar, 16 2015	FG	General correction	All

3. Description

This procedure covers the second phase of DarkSide 50 that involves removing the normal Argon used in the detector and refilling the detector using low background Under Ground Argon (UAr).

For doing that, an additional heated getter gas purification system has been added to DS50 for the final purification of the UAr gas as it is transferred into the detector (up-to-date P&ID and drawing are on docDb).

There are several steps that need to be accomplished in order to prepare DS50 for the use of UAr and prevent any accidental loss of this precious gas:

1) Leak test of entire recovery and getter system.
2) Test recovery system (condensing).
3) Test compressing the gas from recovery system in to empty cylinders.
4) Getter System Test
5) Recovering DS50 high purity Ar and compressing it into cylinders for storage
6) System Maintenance (Changing vacuum pumps and gauges)
7) Cleaning System (evacuation and backfilling with N2)
8) Filling DS50 with UAr above atmospheric pressure
9) Filling DS50 with UAr at sub atmospheric pressure (if required)

4. Plants in Use and Reference Documents

The plants in use are the Getter System placed on the ground flour (below the blue stairs), the Cooling Tower (in to CRH), the Ar Recovery System and the DS Detector.

The reference source for the P&ID is the DarkSide DocDB Document.

5. Distribution list

- ✓ DarkSide Collaboration through the database Darkside-DocDb.fnal.gov
- ✓ ~~LNGS Prevention and Protection Service (SPP).~~
- ✓ ~~LNGS Environment Service.~~
- ✓ ~~LNGS Technical Division.~~
- ✓ ~~LNGS Research Division.~~
- ✓ ~~LNGS Directorate.~~

6. References

- ✓ D. Lgs. 81/08 and later modifications.
- ✓ D. Lgs. 334/99 and later modifications.
- ✓ LNGS Safety Procedure (Sistema Gestione Sicurezza - SGS).
- ✓ LNGS Enviromental Procedure (Sistema Gestione Ambientale - SGA; PG. 06.01 “Gestione dei rifiuti”).

7. Hazards of Unit Operations & Safety Instructions

A. Introduction

The above mentioned Experiment plants should be operated only with management approval, and only at agreed scheduled times, with authorized collaboration personnel, and only for the specific operations planned by the DS Collaboration.

The activity requires the minimum of #2 authorized persons. Authorized persons are the operators which have been trained for technical specific purpose and safety.

For each shift of operations, check that there are no scheduled access restrictions (e.g. construction work or bad weather), or planned interruptions in electrical, telephone, ventilation.

The details of the operation will be reported in the in the DS-Elog and on the Operations Paper Logbook (present in the Bx/DS Control Room).

Check that the Borexino/DarkSide safety manual is available underground.

B. Chemical Safety

During the process will be used Argon and Nitrogen. A summary of the hazards, precautions and first aid to be used for each of these chemicals is listed below.

• Exposure limits and definitions

✓ **PEL.** U.S. Government OSHA Permissible Exposure Limits. PEL and TLV refers to airborne concentrations measured in the breathing zone by appropriate sampling techniques.

✓ **ACGIH.** American Conference of Governmental Industrial Hygienists.

✓ **TLV-TWA.** The time-weighted average concentrations for a normal 8 hour workday or 40 hour work week, to which nearly all workers may be repeatedly exposed, day after day, without adverse effect.

✓ **TLV-STEL.** The short term exposure limit (TLV-STEL) is the maximum concentration to which workers can be exposed for a period of up to 15 minutes continuously without suffering from (1) irritation, (2) chronic or irreversible tissue change, or (3) narcosis of sufficient degree to increase accident proneness, impair self-rescue, or materially reduce work efficiency, provided that no more than four excursions per day are permitted, with at least 60 minutes between exposure periods, and provided that the daily TLV-TWA also is not exceeded. The STEL should be considered a maximum allowable concentration or absolute ceiling not to be exceeded at any time during the 15 minute excursion period.

✓ **TLV-C.** The threshold limit value - ceiling concentration that should not be exceeded even instantaneously. For most substances, e.g., irritant gases, only one category, the TLV-Ceiling, may be relevant. For other substances, either two or three categories may be relevant depending upon their physiologic action.

It is important to observe that if any one of these three TLV's is exceeded, a potential hazard from that substance is presumed to exist. The TWA-STEL should not be used as engineering design criterion or considered as an emergency control of health hazards and should not be used as fine lines between safe and dangerous concentrations.

• Definition of Species

✓ **Argon (CAS: 7440-37-1).** Argon gas is moved from the Argon rack through the above mentioned plants to DS detector as Liquid Argon. In hall C is not considered to be a hazard, except in confined areas where it could displace air and suffocate the inhabitants as in CRH (Clean Room Hanoi).
Refer to their MSDSs for further hazardous and safety measures available on DarkSide DocDB Document 301.

✓ **Nitrogen (CAS: 7727-37-9).** Nitrogen gas is used for internal gas blanketing, instrument control. Liquid Nitrogen is used for refrigerate and liquify Argon gas.
Refer to their MSDSs for further hazardous and safety measures available on DarkSide DocDB Document 301.

C. First Aid

In case of contact with the substances acts as below:

✓ If inhaled
If breathed in, move person into fresh air. If not breathing give artificial respiration. Consult a physician.

✓ Refer to their MSDSs for further hazardous and safety measures available on DarkSide DocDB Document 301.

D. Leaking from Pipes or Equipment and Alarms

In case of gas or liquid leakage, if possible isolate the leaking lines or equipment and shutting down plants as necessary.

All the safety and environmental rules and procedures valid for LNGS, along with all general LNGS guidelines and italian laws, are extended to this procedure.

In case of alarm follow the Emergency Plan of LNGS.

E. Recommended major safety recommendation and equipment

✓ **Check ventilation plant** is working properly before starting any operation.
✓ **Check Oxygen Alarm** before starting any operation.
✓ **Portable Oxygen Sensor** are mandatory during the operations inside the CRH.

F. General Safety Conditions

The risk of asphyxiation is mitigated by the fact that part of the operations are performed in the Hall C of the underground laboratories (approx. 100×20×18 meters L×W×H). The quantity in use is very limited with respect to the volume of Hall C. In addition, the forced ventilation in Hall C operates at around 8,000 m^3 /h.

The risk inside the CRH is mitigated by the presence of fixed and portable oxygen sensors.

TBC

8. Procedure

The procedure consists of the following steps:

1) Leak test of entire recovery and getter system.
2) Test recovery system (condensing).
3) Test compressing the gas from recovery system in to empty cylinders.
4) Getter System Test.
5) Recovering DS50 high purity Ar and compressing it into cylinders for storage.
6) System Maintenance (Changing vacuum pumps and gauges).
7) Cleaning System (evacuation and backfilling with N_2).
8) Filling DS50 with UAr.

Important note: Maximum Allowable Working Pressure (MAWP) of sub systems:
High pressure piping MAWP: 3000 psig.
Low pressure piping MAWP: 100 psig.
DS-50 recovery system MAWP: 1.29 barg (19.2 psig).

New System Configuration

Fig. 1 Illustrates a temporary system setup that will be used for steps 1 through 5.

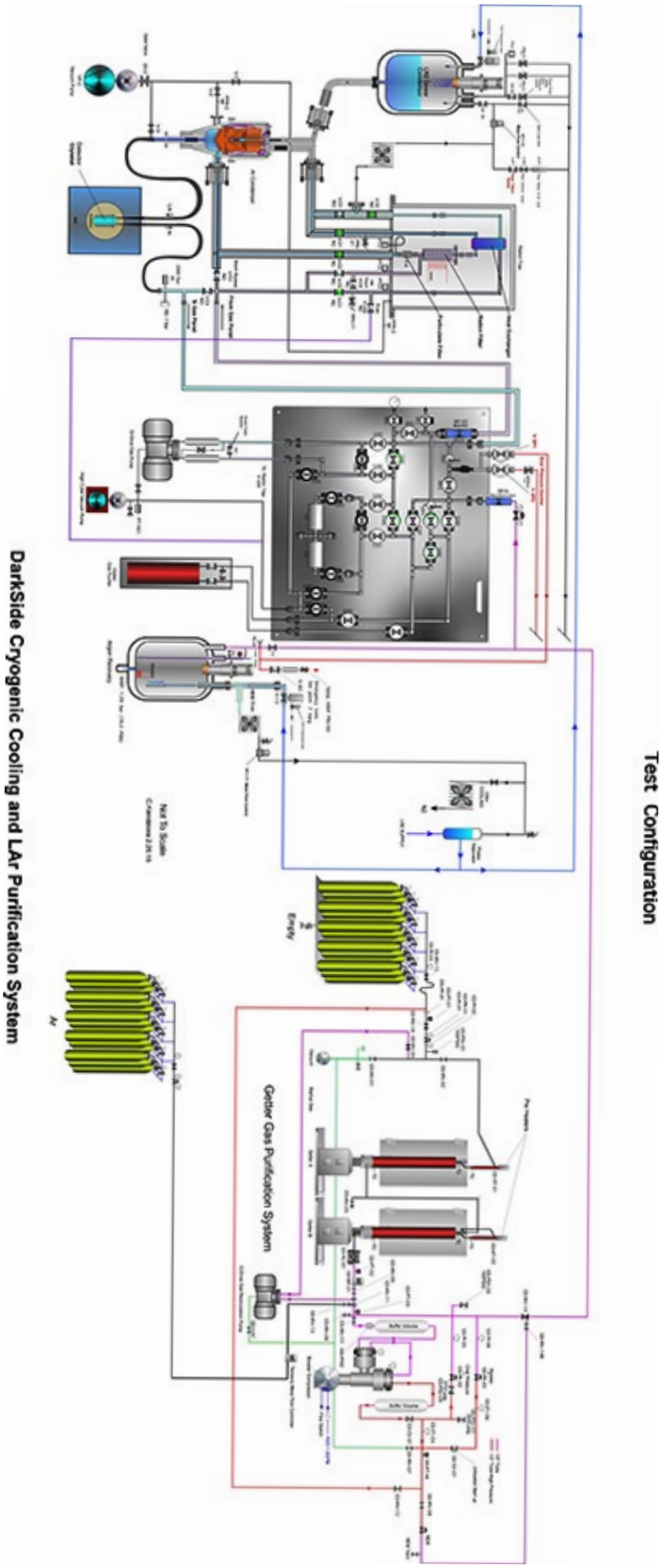

Fig. 1: (System Configuration)

I. Leak Test

The getter and recovery systems must be evacuated and leak checked using a helium mass spectrometer with a sensitivity greater than 1X10-9. Any detectable leak is not acceptable. *Completed*

II. Recovery System

Will be a set of tests that will be performed to understand and verify the performance of the Recovery System. This includes a Condensing Capacity Test of each of the condensers, a Power Failure Test using the LN_2 condenser as an alternative condenser, Emergency Pressure Limit Test and a compressor test that will compress the argon into high pressure cylinders.

Pressure Safety Limit Test

The pressure of 1.29 barg is the Maximum Working Pressure (structural limit) of the recovery dewar. The recovery system has a pressure limit rupture disk that is set for 1.13 bar. The rupture disk is the final safety device in case all else fails. In addition to the rupture disk there is a pneumatic valve with a set point of .7 barg to open and .6 to close. This valve will open and close venting off small quantities of gas keeping the pressure below the rupture disk limit. During normal operation the cryo coolers will keep the pressure in the recovery system below these pressure limits. *Completed*

Condensing Performance Tests

Normal argon will be fed into the system through MV-06 using a temporary mass flow controller. The flow of argon will bypass the getter and compressor circuits and go straight to the recovery system. At the recovery system the argon will be condense and liquid will be accumulated. The LN2 condenser will be tested first to understand it's performance. Because of it's unique custom design, little is known about it's performance. Second, the AL 300 cryo cooler performance will be verified.

Power Failure Test

Knowing that the entire inventory of UAr may someday be located in the recovery system, it's important that we test for a power failure condition. In this condition the LN2 condenser should go into operation. This condition needs to be tested and verified that the recovery system can maintain it's self even during a complete power blackout. *50% Completed*

Compressor Test

Once a significant amount of argon has been condensed in the recovery system, the process will be reversed. A small heater (Omega KHLV-105/10) is now located at the bottom of the dewar of the recovery system. The heater will be used to boil off some liquid that will feed the compressor with a gas. The compressor will compress the gas into the empty set of cylinders. It's important that the heater power not boil off the gas at a rate that

exceeds .9 barg in the dewar. There is a software interlock that will turn off the heater in the event the .9 barg is reached.

Preparation for DS50 Ultra Pure Argon Recovery

After all of Recovery System commissioning tests have been completed the Normal Argon will need to be vented out of the high pressure cylinders and the entire system including the high pressure cylinders must be evacuated to remove any standard argon left in the system. The normal argon cylinders of argon can be removed and valve GS-MV-13 can be closed.

III. Recovery of DS50 Ultra Pure Argon

The high purity argon that is presently in DS50 will be removed by condensing the argon in the recovery system using the recovery system cryo cooler. It will then be compressed into evacuated cylinders by the high pressure compressor. The goal would be to condense the gas in the recovery system while compressing the gas in the cylinders.

The transfer of DS50 ultra high purity gas will take place as illustrated in fig 2.

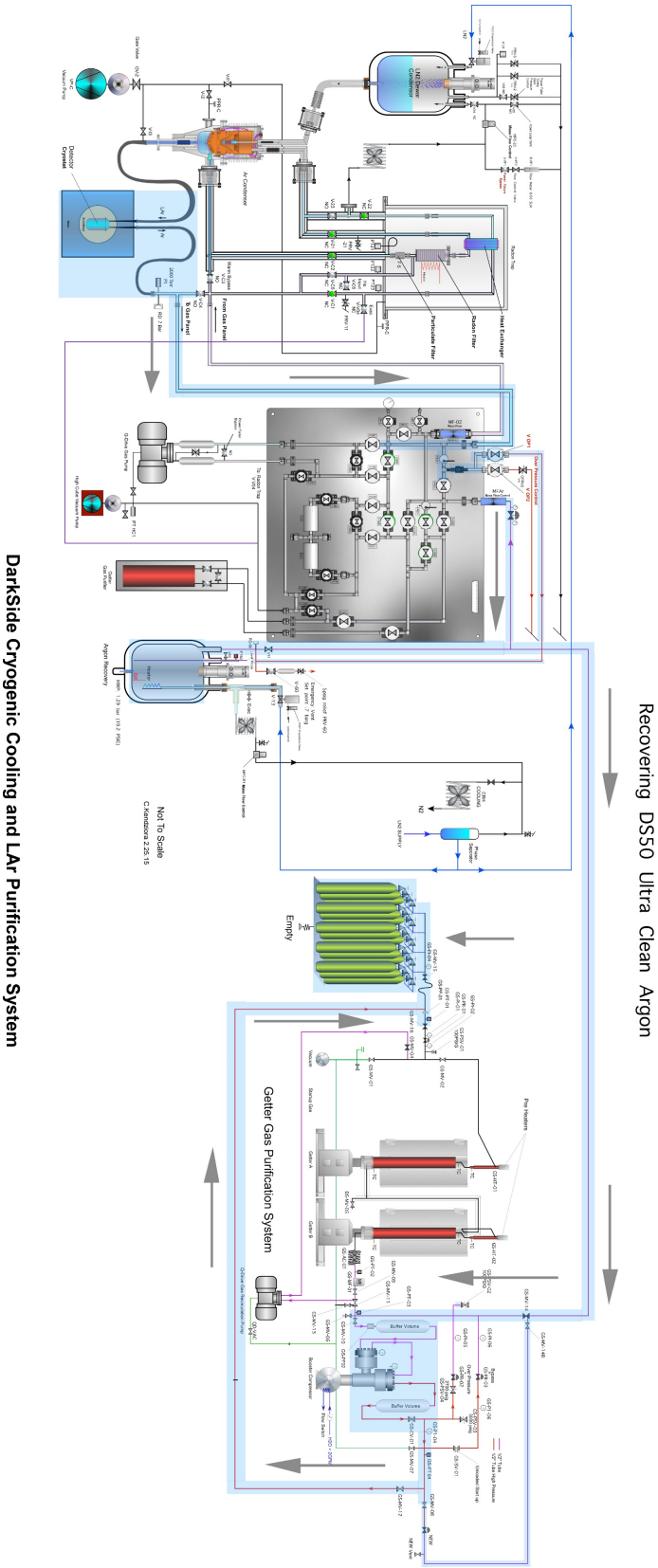

Fig. 2: (DS50 Ultra Pure Argon Recovery)

The “Empty” rack which was temporarily used during the recovery system tests will need to be located in the position where the Normal Argon rack was removed from and completely evacuated.

Near the end of the transfer of the gas when there is almost no liquid argon in DS50 the cooling system will be put back in a normal operating condition and a power failure test will be performed on the control system to verify that it can successfully reboot and load the operating control software correctly without any issues. Performing this test with very little argon left in the detector minimizes the potential loss of any of DS50 high purity argon but yet allows us to see what will happen during a total power failure in a normal operating condition.

Once the power failure test of the control system has been completed the rest of the ultra clean argon will be recovered into the recovery system and then compressed into the empty high pressure cylinders for storage. There will be a scale located under the rack of these cylinders that will be used to weigh the content of the gas from DS50. The amount of weight measured will be the quantity of UAr needed to fill DS50 as it was.

Operating Procedures

- **Prerequisites**

  ☐ The present procedures approved by DS50 management.
  ☐ ……
  ☐ …..
  ☐ …..

- **Alignment check list**

  Getter System
  ☐ GS-MV-14, GS-MV-10, GS-CV-01, GS-MV-12 and GS-MV-15 opened (Highpressure line).
  ☐ GS-MV-08, GS-MV-06, GS-MV-11, GS-SV-01, GS-PR-0, GS-MV-19 and GS-MV-07 closed.
  ☐ GS-PR-02, GS-PR-03, GS-PR-04 regulated and GS-MV-08 opened (low pressure line).
  ☐ …..

  Ar Recovery
  ☐ V-11 opened.
  ☐ …..
  ☐ ……
  ☐ ……

  Cooling Tower
  ☐ Regulate PR-1 at XXX
  ☐ V-G07, V-G08, V-G05, V-G11, V-GG2, V-GG3, V-G12, V-G03 opened.
  ☐ V-C4, V-G04, V-S04, V-G09, V-G10, V-G06, V-G01 closed.
  ☐ ……

Operative procedures

- ☐ Regulate XXXX
- ☐ Turn XXXX on/off
- ☐ Doing something??

Getter Controls Test

Once the leak check of the getters has been completed. A closed loop circulation test using the Q drive should be performed using normal argon as illustrated in Fig. 6. This test will be to verify that all the instrumentation and controls work correctly using a small amount of DS ultra pure argon.

System Maintenance

After the detector has been warmed up and empty of Ultra Pure Argon, the insulating vacuum system should be turned off and the system maintenance should be performed. This incorporates changing the fore pump of the main turbo in the cooling tower and the bad vacuum readouts in the vacuum system.

This also includes changing all of the plastic fittings and tubing. With the entire system warm provides the opportunity for all the frozen gas impurities to be pumped out.

System Cleaning

The entire cooling tower system including the detector needs to be evacuated and backfilled (X3) with N_2 to displace any Argon that may contain Ar39. All trapped volumes of argon in the system need to be identified, evacuated and purged except for the krypton source. The krypton source can be evacuated separately from the rest of the system avoiding the possibility of putting krypton in the system at this time. There is concern that the TPB will evaporate and there is some worry about the PMT electrical solder joints failing if a full vacuum is achieved in the detector. Therefor the DS50 detector should only be evacuated to 1x10-1 mbar and then immediately backfilled with N_2 to one atmosphere three times. It should be left with N_2 at atmosphere and isolated by closing valves VC-2, VC-3 VC-4 and MF2 until the UAr is brought online. The insulating vacuum will be maintained and the detector will be left below freezing point of water.

IV. Filling DS50 with UAr (above atmospheric pressure)

UAr is a rare precious gas that requires great care in handling to prevent any possible losses or contamination.

The filling of DS50 with UAr needs to be configured as illustrated in Fig 5.

The cylinders that contain the DS50 ultra pure argon needs to be removed from the system. The manifold for connecting all of the cylinders of UAr need to be put in place and leak checked.

The entire system up to and including the detector needs to be evacuated and purged with N_2 as previously mentioned. The getter system must

be fully operational before any UAr is added to the system by adding a small amount of UAr to the getter circulation circuit as illustrated in Fig. 6, then recirculating the gas in a closed loop using the Q drive pump until the getters are at 400 °C for 4 hours as illustrated in Fig. 6.

Once the getters are up to 400 °C for four hours, the UAr gas should be allowed to flow through the getters into the rest of the system up to the MF2. The detector should then be evacuated to a full vacuum of 10-2 mbar and then immediately backfilled with UAr through MF2 (Is it GS-MF-02?, or is it GS-MF-01?).

The cooling tower needs to be brought on line and the full inventory of UAr be condensed into the detector. The empty cylinders of UAr will be permanently left in place in the event the UAr needs to be transferred back into the original cylinder rack for storage long term.

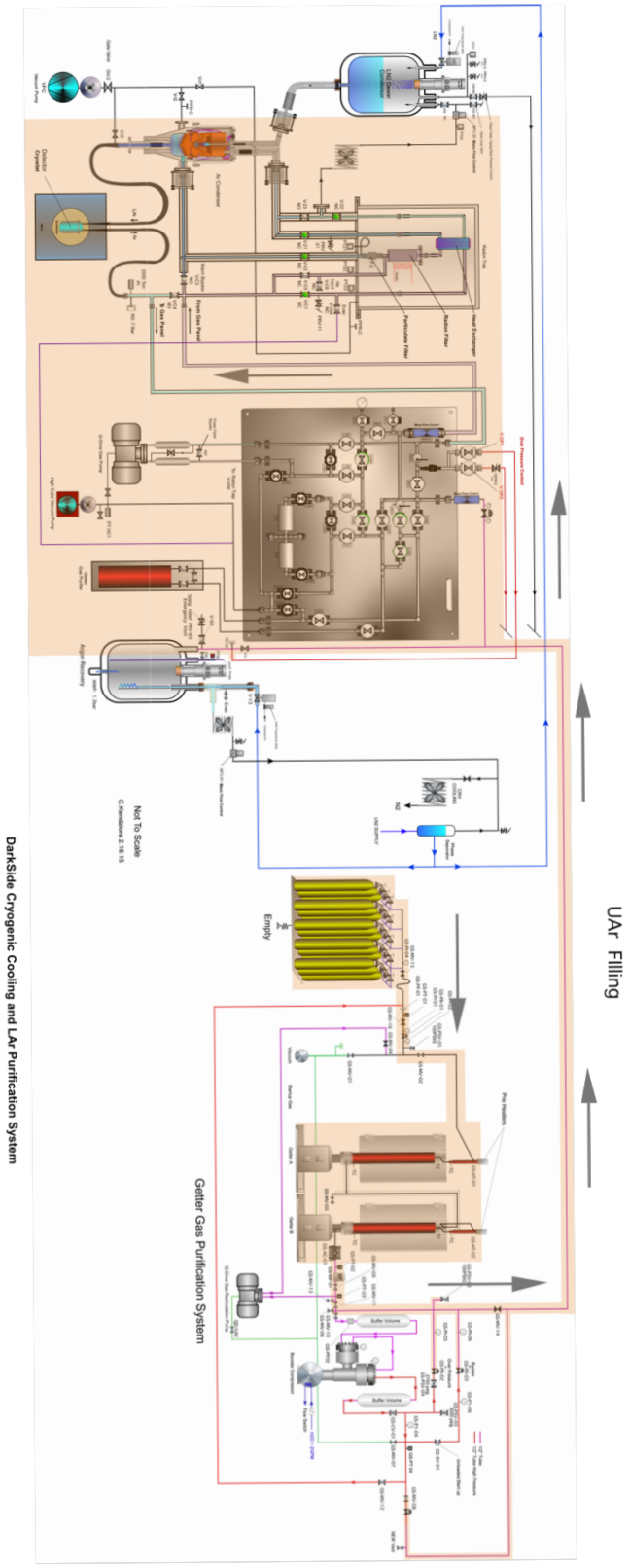

Fig. 5: (Filling Configuration)

Operating Procedures

- **Prerequisites**

☐ The present procedures approved by DS50 management.
☐ System leak tested.
☐ System evacuated and purged.
☐ Getter System fully operating and recirculating with a small amount of UAr in close loop using the Q drive for 4 hours up to 400 °C.
☐ Getter System reach 400 °C.

- **Alignment check list**

Getter System
☐ GS-PR-01 regulated at XXX.
☐ GS-MV-02 opened.
☐ GS-MV-09 and GS-MV-11 opened.
☐ GS-MV-13, GS-MV-10, GS-MV-06, GS-PR-02, GS-PR-03 closed.
☐ GS-MV-14 opened.

Ar Recovery
☐ V-11 closed.
☐ ……
☐ ……

Cooling Tower
☐ XXX .
☐ ……
☐ ……

Operative procedures

☐ GS-MF-02 regulated (or opened?).
☐ Q drive on ……
☐ ……

9. Operations and Shifts Requirements

During the activity will be present at least:

- ✓ Three DarkSide operators

Leading personnel involved in the operation and contact numbers:

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